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Indian Standard

NEW INSULATING OILS — SPECIFICATION

(Fourth Revision)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Fluids for Electrotechnical Applications Sectional Committee had been approved by the Electrotechnical Division Council.

This standard, first published in 1953, was based on BS 148 : 1951 'Insulating oil for transformers and switchgear', issued by the British Standards Institution. It was revised in 1963 to bring the test methods in line with the practices in vogue. The second revision was undertaken in 1972 mainly to include oxidation test as given in IEC Pub 296 (1982) 'Specification for unused mineral insulating oils for transformers and switchgear'. The third revision was undertaken in 1983 to include an ageing test based on ASTMD 1934 : 1968 'Standard method of test for oxidative ageing of electrical insulating petroleum oils by open beaker method', issued by the American Society for Testing and Materials.

This fourth revision includes amendments issued since the adoption of third revision and also a new method of test adopted for detection of oxidation inhibitor.

In preparing this revised standard, considerable assistance has been derived from IEC Pub 296 (1982) 'Specification for unused mineral insulating oils for transformers and switchgear', issued by the International Electrotechnical Commission (IEC). BS 148 : 1984 'Specification for insulating oils for transformers and switchgear', issued by the British Standards Institution.

Where by arrangement between the purchaser and the supplier, an oil containing an oxidation inhibitor or other additive, that is, an inhibited oil is supplied, the base oil used shall comply with the requirements of this standard.

Separate standard, IS 12463 : 1988 'Specification for inhibited mineral insulating oils' has since been brought out for inhibited mineral insulating oils.

For the maintenance and supervision of insulating oils conforming to this specification and used in transformers, switchgear and certain other similar oil immersed equipment, reference shall be made to IS 1866 : 1983 'Code of practice for maintenance and supervision of mineral insulating oil in equipment (*second revision*)'.

For the insulating oil conforming to this specification, the typical (approximate) values (all relating to 60°C) of a few properties other than covered in Table 1 are given below as a guide. Figures in parenthesis indicate the approximate temperature coefficient (per degree Celsius for the property concerned):

Coefficient of expansion over the normal range of operating temperature	0·000 78/°C
Permitivity	2·2 (−0·001)
Specific heat	2·06 kJ/kg°C (0·003 8)
Thermal conductivity	0·12 W/m°C

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard .

Indian Standard

NEW INSULATING OILS — SPECIFICATION

(Fourth Revision)

1 SCOPE

1.1 This standard prescribes the requirements of new insulating oil of petroleum origin suitable for use as an insulating and heat transfer medium and are quenching medium for power and distribution electrical apparatus, such as transformers, switchgears, capacitors and allied equipments.

1.1.1 The oils covered by this standard are low viscosity type completely free from additives.

1.2 This standard does not apply to:

a) inhibited oils (refers to IS 12463 : 1988);
b) oils required for cables and switchgear requiring high viscosity oils or for special impregnation purposes; and
c) synthetic dielectric liquids.

2 REFERENCES

2.1 The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 TERMINOLOGY

3.0 For the purpose of this standard, the following definitions shall apply.

3.1 Flash Point

The temperature at which the oil gives off so much vapour that this vapour, when mixed with air, forms an ignitable mixture and gives a momentary flash on application of a small pilot flame under the prescribed conditions of test.

3.2 Pour Point

The lowest temperature expressed as a multiple of 3°C at which the oil is observed to flow when cooled and examined under prescribed conditions.

3.3 Total Sludge Value

The percentage by weight of insoluble matter formed when the oil is heated and oxidized under specified conditions and subsequently diluted with n-heptane.

3.4 Neutralization Value

3.4.1 *Total Acidity*

It is the measure of free organic and inorganic acids present together and is expressed as milligrams of potassium hydroxide required to neutralize the total free acids in one gram of the oil.

3.4.2 *Inorganic Acidity*

It is the measure of inorganic acids present and is expressed as milligrams of potassium hydroxide required to neutralize these acids in one gram of the oil.

3.5 Electric Strength (Break Down Voltage)

The voltage at which the oil breaks down when subjected to an ac electric field with a continuously increasing voltage contained in a specified apparatus. The voltage is expressed in kV (rms).

3.6 Specific Resistance (Resistivity)

It is the ratio of the dc potential gradient in volts per centimetre paralleling the current flow within the specimen, to the current density in amperes per square centimetre at a given instant of time and under prescribed conditions. This is numerically equal to the resistance between opposite faces of a centimetre cube of the liquid. It is expressed in ohm-centimetre.

3.7 Dielectric Dissipation Factor (Tangent Delta)

It is the tangent of the angle (delta) by which the phase difference between applied voltage and resulting current deviates from $\pi/2$ radian, when the dielectric of the capacitor consists exclusively of the insulating oil.

3.8 Interfacial Tension

It is the force necessary to detach a planar ring of platinum wire from the surface of the liquid of higher surface tension that is upward from the water-oil surface. It is expressed in N/m.

3.9 SK Value

The SK value is the increase in the volume of concentrated sulphuric acid on adding a given sample in the prescribed apparatus following defined procedure.

3.10 Repeatability

A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

3.11 Reproducibility

A quantitative measure of variability associated with operators working in different laboratories, each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run only in one case in twenty in the normal and correct operation of the test method.

4 COMPOSITION

The oil shall be pure hydrocarbon mineral oil, without any additive, clean and sufficiently free from moisture or other foreign matter likely to impair its properties.

5 CHARACTERISTICS

The characteristics of the oil when it is sampled (*see* 7) at the manufacturer's work and/or at the point of delivery and tested in accordance with the methods referred to in Table 1 shall comply with the requirements specified in Table 1.

6 PACKING

6.1 The oil may be delivered in perfectly clean steel drums of 210 litres nominal capacity conforming to Type A or Type B in IS 1783 (Part 1) : 1983 and IS 1783 (Part 2) : 1988 respectively. The drum shall be coated from inside with suitable coating (for example, epoxy lacquer of phosphate) resistant to insulating oil. The outside surface of the drum may be coated with suitable primer and finishing paint, or hot dip galvanized according to IS 4759 : 1984 for protection against atmospheric corrosion. If primer is not used then, the outside surface of the drum shall be suitably treated (say by solvent) and painted by a suitable paint for protection against atmospheric corrosion. The drum shall be effectively sealed immediately after filling the oil to avoid ingress of moisture.

6.2 Oil may also be delivered in a suitable type of high density polyethylene (HDPE) barrel subject to agreement between the purchaser and the manufacturer.

6.3 Oil may also be delivered in road or rail tank wagons specially cleaned and reserved for this purpose and shall be suitably sealed so as to avoid ingress of moisture.

NOTES

1 Clean drums specially reserved should be used for the purpose of delivering and storing insulating oil.

2 Oil in drums cannot be expected indefinitely to retain the electrical characteristics shown at the time of filling and these are likely to deteriorate during storage.

7 SAMPLING

Sampling of the oil shall be done in accordance with IS 6855 : 1973.

8 MARKING

8.1 Each drum shall be indelibly marked with the following:

a) Manufacturer's name or trade-mark,

b) Quantity in litres,

c) New mineral insulating oils, and

d) Identification in code or otherwise to enable the date and lot of manufacture to be traced back to the factory records.

8.1.1 The containers may also be marked with the Standard Mark.

8.2 When supply of insulating oil is made in road or rail tank wagons, it shall be accompanied with a certificate giving information as required in **8.1.**

9 TESTS

9.1 The tests shall be carried out in accordance with the test methods referred to in Table 1.

9.1.1 *Quality of Reagents*

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

Table 1 Schedule of Characteristics

(*Clauses* 5.1 *and* 9.1)

Sl No.	Characteristics	Requirement	Method of Tests	Remarks
(1)	(2)	(3)	(4)	(5)
i)	Appearance	The oil shall be clear and transparent and free from suspended matter or sediments	A representative sample of the oil shall be examined in a 100 mm thick layer at 27°C	—
ii)	Density at 29·5°C, *Max*	0·89 g/cm³	IS 1448 [P : 16] : 1977	*See* NOTE 1
iii)	Kinematic viscosity, *Max*, at		IS 1448 [P : 25] : 1976	—
	a) 27°C	27 cSt		
	b) 40°C	Under consideration		
iv)	Interfacial tension at 27°C, *Min*	0·04 N/m	IS 6104 : 1971	—
v)	Flash point Pensky-Marten (closed), *Min*	140°C	IS 1448 [P : 21] : 1970	—
vi)	Pour point, *Max*	−6°C	IS 1448 [P : 10] : 1970	—
vii)	Neutralization value			
	a) Total acidity, *Max*	0·03 mg KOH/G	IS 1448 [P : 2] : 1967	Alcoholic potassium hydroxide solution of 0·02 N should be used in place of 0·1 N indicated in test method
	b) Inorganic acidity/ alkalinity	Nil	do	
viii)	Corrosive sulphur	Non-corrosive	Annex B	—
ix)	Electric strength (breakdown voltage)		IS 6792 : 1972	—
	a) New unfiltered oil, *Min*	30 kV (rms)		
	b) After filtration, *Min*	If the above value is not attained, the oil shall be filtered 60 kV (rms)		*See* Note 2
x)	Dielectric dissipation factor (tan δ) at 90°C, *Ma*	0·002	IS 6262 : 1971	*See* Note
xi)	Specific resistance (resistivity)		IS 6103 : 1971	*See* Note 2
	a) At 90°C, *Min*	35×10^{12} ohm-cm		
	b) At 27°C, *Min*	$1\,500 \times 10^{12}$ ohm-cm		
xii)	Oxidation stability		Annex C	—
	a) Neutralization value after oxidation, *Max*	0·4 mg KOH/g		
	b) Total sludge, after oxidation, *Max*	0·1 percent by weight		

Table 1 (*Concluded*)

(1)	(2)	(3)	(4)	(5)
xiii)	Ageing characteristics after accelerated ageing (open beaker method with copper catalyst)	—	IS 12177 : 1987 Method A	—
	a) Specific resistance (resistivity)		IS 6103 : 1971	
	1) at 27°C, *Min*	$2{\cdot}5 \times 10^{12}$ ohm-cm		
	2) at 90°C, *Min*	$0{\cdot}2 \times 10^{12}$ ohm-cm		
	b) Dielectric dissipation factor (Tan δ) at 90°C, *Max*	0·20	IS 6262 : 1971	
	c) Total acidity, *Max*	0·05 mg KOH/g	IS 1448 [P : 2] : 1967	
	d) Total sludge, *Max*	0·05 percent by weight	Annex A of IS 12177	
xiv)	Presence of oxidation inhibitor	The oil shall contain anti-oxidant additives	IS 13631 : 1992	*See* Note 3
xv)	Water content, *Max*	50 ppm	IS 13567 : 1992	—
xvi)	SK value	Under consideration	Annex D	—

NOTES

1 Density of the oil may be measured at ambient temperature and converted to 29·5°C using the following equation:

$$29{\cdot}5\ \rho = \rho_t\ [\,1 + X(t - 29{\cdot}5)\,]$$

where

t = ambient temperature (in °C),

ρ^t = density measured at temperature t, and

X = correction factor (equal to 65×10^{-5} approximately).

2 As a consequence of the tendency for water absorption to occur due to breathing on storage even when drums are sealed the oil shall be filtered to remove moisture and particulate contaminants present in the original sample before the test as follows:

'A sufficient quantity of oil is heated to 90 ± 2°C, then filtered hot under vacuum corresponding to an absolute pressure of about 2·5 kPa through a sintered glass filter of porosity grade 4'.

A portion of the filtrate is cooled in a desiccator and used immediately to measure electric strength, if required, and specific resistance at 27°C. The remaining hot filtrate is immediately used for measuring dielectric dissipation factor at 90°C and specific resistance at 90°C.

3 For both phenol and amine types of indicators, qualitative method as per Annex D shall be adopted. In case of ambiguity (marginal cases) in finding the intensity of colour, a quantitative method as given in Annex D shall be adopted. Value of 0·5 (*Max*) shall be treated as absence of DBPC-Phenolic type inhibitor (Quantitative method for amine is under consideration).

ANNEX A

(*Clause* 2.1)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*	*IS No.*	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)	6103 : 1971	Method of test for specific resistance (resistivity) of electrical insulating liquids
1448 [P : 2] : 1967	Methods of test for petroleum and its products: Part 2 Acidity (*first revision*)	6104 : 1971	Method of test for interfacial tension of oil against water by the ring method
1448 [P : 10] : 1970	Methods of test for petroleum and its products : Part 10 cloud point and pour point (*first revision*)	6262 : 1971	Solvent extracted undecorticated safflower oilcake (meal) as livestock feed ingredient (*first revision*)
1448 [P : 16] : 1990	Methods of test for petroleum and its products : Part 16 Density of crude petroleum and liquid petroleum products by hydrometer method (*third revision*)	6272 : 1971	Metal polishes (special)
		6792 : 1972	Method for determination of electric strength of insulating oils
		6855 : 1973	Method of sampling for liquid dielectric
1448 [P : 25] : 1976	Methods of test for petroleum and its products : Part 25 Determination of kinematic and dynamic viscosity (*first revision*)	12177 : 1987	Methods of test for oxidative ageing of electrical insulation of petroleum oils by the open beaker method
1783 (Part 1) : 1983	Drums, large, fixed ends: Part 1 Grade A drums (*second revision*)	12463 : 1988	Inhibited mineral insulating oils
1783 (Part 2) : 1988	Drums, large, fixed ends: Part 2 Grade B drums (*third revision*)	13567 : 1992	Determination of water in insulating liquids and in oil-impregnated paper and press board by automatic coulometric Karl Fischer titration — Method of test
2832 : 1964	Waterproof silicon carbide paper (withdrawn)		
4759 : 1984	Hot-dip zinc coatings on structural steel and other allied products (*second revision*)	13631 : 1992	Method of test for detection and determination of antioxidant additives in insulating oils.

ANNEX B

[*Table* 1, *Item* (viii)]

METHOD OF TEST FOR CORROSIVE SULPHUR IN ELECTRICAL INSULATING OILS

B-1 GENERAL

B-1.1 This method covers the detection of corrosive sulphur compounds in electrical insulating oils of petroleum origin.

B-1.2 Mineral insulating oils may contain substances that cause corrosion under certain conditions of use. This test is designed to detect objectionable quantities of free sulphur and corrosive sulphur compounds by subjecting copper to contact with oil under prescribed conditions.

B-2 SIGNIFICANCE

B-2.1 In most of their uses insulating oils are continuously in contact with metals that are subject to corrosion. Since the presence of detrimental corrosive sulphur compounds will result in deterioration of these metals to an extent dependent upon the quantity and type of corrosive agent and the time and temperature factors, the detection of these undesirable impurities, even though not in terms of quantitative values, is a means for recognizing the hazard involved.

B-3 APPARATUS

B-3.1 Bath

A hot-air oven or oil bath provided with suitable means of heating to, and controlled at, 140 +2°C. A circulating hot-air oven is preferred.

B-3.2 Containers

Conical flask of chemically resistant glass, 250 ml capacity, capable of holding 270 to 280 ml when filled completely to the stopper. Bottles of such capacity are required in order to allow sufficient space for expansion of the oil.

B-3.3 Electrolytic Copper Sheet

0·127 to 0·254 mm in thickness.

B-3.4 Polishing Material

Polishing material consisting of 240 grit silicon carbide paper (IS 2832 : 1964) or cloth, and also 150 mesh silicon carbide grains and cotton wool.

B-4 REAGENTS

B-4.1 Acetone

Chemically pure.

B-4.2 Ether

Chemically pure.

B-4.3 Nitrogen Gas

Commercial cylinders of nitrogen gas are satisfactory for this purpose.

B-5 PREPARATION OF APPARATUS

B-5.1 Bottles shall be chemically cleaned. Clean the bottles with suitable solvents to remove oils; then wash the bottles with phosphate-type cleaning powder. Rinse with tap water, then with distilled water, and dry in an oven.

B-5.2 Cut a strip of copper 6 mm wide and 25 mm long (*see* Note) and remove blemishes from surfaces with the 240-grit silicon carbide paper. These strips may be stored in sulphur-free acetone at this point for future use. Do the final polishing of the strip by removing it from the acetone, holding it in the fingers protected with ashless filter paper, and rubbing with 150 mesh silicon carbide grains picked up from a glass plate with a pad of absorbent cotton moistened with a drop of acetone. Wipe the strip with fresh pads of cotton and subsequently handle only with stainless steel forceps (do not touch with the fingers). Rub in the direction of the long axis of the strip. Clean all metal dust and abrasive from the strip, using successive clean cotton pads until a fresh pad remains unsoiled. Bend the clean strip in a V-shape at approximately a 60° angle and wash successively in acetone, distilled water, acetone, and ether. Dry in an oven for only a few minutes and immediately immerse in the prepared sample.

NOTE — It has been found convenient to polish a larger piece of copper from which, after the final polishing several strips of the proper size may be cut.

B-6 PROCEDURE

B-6.1 The oil to be tested, in general, should be used as received and should not be filtered through paper. Promptly place the prepared copper strip in a clean 250 ml bottle to which 250 ml of the oil has been added. Place the bent copper strip standing on its long edge so that no flat surface lies along the glass bottom of the vessel. Lubricate the ground-glass stopper with a small amount of the sample. Bubble nitrogen through the oil in the bottle by means of a glass tube connected to the reduction or needle valve of the cylinder (rubber connections should be sulphur-free) for one minute, and quickly put the stopper loosely in place.

B-6.2 Place the stoppered bottle (immersed to the neck in the event an oil bath is employed) in the oven at 140°C. When the oil in the flask has reached approximately 140°C, the stopper may be tightened more firmly. Remove the bottle after heating for 19 hours at 140 ± 2°C. Carefully take the copper strip from the flask and wash with acetone or other suitable solvent to remove all sticking oil.

B-6.3 To inspect, hold the test strip in such a manner that light reflected from it at an angle of approximately 45 degrees will be observed.

B-7 INTERPRETATION

B-7.1 The oil shall be classified as corrosive or non-corrosive in accordance with Table 2.

Table 2 Copper Strip Classification
(*Clause* B-7.1)

Classification	Description
Non-corrosive	Orange, red, lavender, multicoloured with lavender blue and/or silver overlaid on claret red, silvery, brassy or gold, magenta overcast on brassy strip, multi-coloured with red and green showing (peacock) but no grey
Corrosive	Transparent black, dark grey or dark brown, graphite or lusterless black, glossy or jet black, any degree of flaking

B-8 REPORT

B-8.1 Report the oil as being corrosive or non-corrosive.

ANNEX C

[*Table* 1, *Item* (xii)]

DETERMINATION OF OXIDATION STABILITY

C-1 GENERAL

C-1.1 The description of this method specifies the conditions for bringing about a series of successive physical and physico chemical reactions of a much more complex nature than measurement of a simple physical property, such as density, viscosity or even the fracture of a metal sample under tension.

C-1.1.1 It is, therefore, essential that such a test should be carried out under the supervision of an expert, both as regards the manner in which it is done and the interpretation of results.

C-1.2 While the test is relatively easy in the case of an already known oil, it may be much more difficult where a new product is concerned. It is known, for example, that certain unrefined oils now contain more sulphur than previously. It may therefore be feared, when examining an unknown oil whose sulphurous constituents are liable to contaminate the metallic catalyst, that abnormal results will be obtained even if every care has been taken to utilize the correct apparatus and to entrust the operations to experienced staff.

C-1.3 The conclusion to be drawn from the above is that special cases will have to be studied separately. The test has been primarily designed for the examination of pure refined petroleum distillates. Its extension to other products is an extrapolation.

C-2 PRINCIPLE

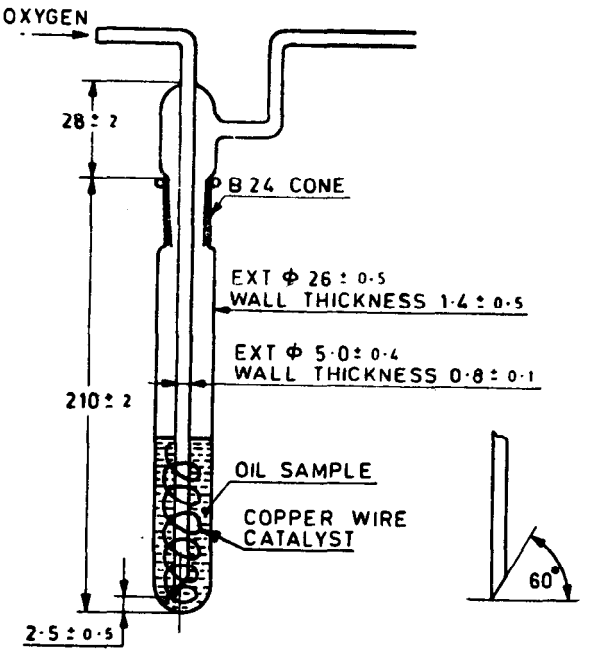
C-2.1 A stream of oxygen is bubbled, through the oil under test, which is maintained for a carefully measured period at a temperature of 100°C in the presence of metallic copper. The degree of oxidation is estimated by determining the amount of sludge and the neutralization value.

C-3 TEST VESSEL

C-3.1 The test vessel shall be a borosilicate or natural glass tube, provided with a B 24 cone, and having the following dimensions:

Overall length	210 ± 2 mm
External diameter	26 ± 0·5 mm
Wall thickness	1·4 ± 0·2 mm
Height of the head	28 ± 2 mm
Oxygen inlet tube:	
a) External diameter	5·0 ± 0·4 mm
b) Wall thickness	0·8 ± 0·1 mm

C-3.2 The test tube is fitted with a Drechsel head to which is attached the inlet tube which extends to within 2-3 mm of the bottom and has its end ground at an angle of 60° to the horizontal axis (*see* Fig. 1).



All dimensions in millimetres.

FIG. 1 TEST VESSEL

C-4 QUANTITY OF OIL

C-4.1 The quantity of oil shall be 25±0·1 g.

C-4.2 The oil, mixed by stirring, is first filtered through a sintered glass filter as described under C-11.1.3.

C-5 REAGENTS

C-5.1 Oxygen

Obtained from liquid air, minimum purity 99·4 percent.

C-5.2 Normal Heptane

Conforming to the following:

a) Density at 20°C : 0·683 80±0·000 15
b) Refractive index at 20°C : 1·387 70± 0·000 15
c) Solidification temperature: −90·71°C, *Min*
d) Distillation: 50 percent recovered 98·427± 0·025°C.

e) Differential : 80 percent recovered minus 20 percent recovered 0·020°C, *Max.*

NOTE — This reagent is identical with n-heptane reference fuel used in testing the octane number of internal combustion engine fuel.

C-5.3 Indicator Solution

A solution of 2 g of alkali blue 6B in 100 ml of pure ethyl alcohol.

C-5.4 Tiration Solution

0·02 N alcoholic potassium hydroxide solution.

C-6 OXYGEN SUPPLY

C-6.1 The oxygen shall be thoroughly dried by passing it through concentrated sulphuric acid and over lime or soda lime, or by any other method of equal or greater efficacy.

C-6.2 A 10 litre flask, acting as a surge vessel, smoothes the oxygen flow, excess of which bubbles through mineral oil contained in a test tube.

The flow rate shall be 1 ± 0·1 l/h.

NOTE — The method used to check the flow rate is left to the discretion of the testing authority.

C-7 METHOD OF CLEANING THE TEST VESSELS

C-7.1 The test-tube shall be chemically cleaned. A method of cleaning which has been found satisfactory is to wash with acetone, distilled water, then with concentrated sulphuric acid which is removed by washing, first with tap water, then with distilled water.

C-7.2 The reagents used shall be of chemically pure grade.

C-7.3 The apparatus is dried in an air-oven at 105-110°C for at least 3 hours, and then allowed to cool to room temperature in a desiccator in which the test-tubes are kept until they are used.

C-8 HEATING ARRANGEMENTS

C-8.1 The test temperature for the oil shall be 100·0±0·5°C.

C-8.2 The temperature of the thermostatic block required to maintain the oil at 100°C governs the temperature of the air above the oil surface. The temperature just below the cover of the test tube shall be not less than 70°C.

C-8.3 A wrought aluminium block provided with holes to accommodate the test vessel, described in C-3, shall be used for heating. Provision should be made in the block for at least four test-tubes (test vessels) and its temperature maintained constant by the use of a contact thermometer. The test-tubes should be inserted as far as possible into the holes. If they cannot be completely inserted, shaft metal collars passing through the insulating cover and surrounding each test-tube will ensure heating over the full length of the tube.

C-9 CATALYST

C-9.1 The metallic copper used as oxidation catalyst consists of a wire of non-annealed commercial electrolytic copper of diameter between 1·00 and 1·02 mm. It is prepared as follows:

a) Immediately before use, the copper wire is polished with No. 00 emery cloth or an equivalent abrasive cloth (*see* Note). All traces of abrasive are removed with a lintless filter paper and then with a dry, lintless cloth.

NOTE — The emery grains used in the manufacture of No. 00 abrasive cloth shall pass completely through 67-mesh/cm sieve.

The mesh characteristics are defined by the following dimensions:

Aperture width	0·089 mm
Wire diameter	0·061 mm

b) A piece of the polished wire 305±1·0 mm long is rolled into a spiral of approximately 20 mm external diameter and 50 mm in length.

c) The spiral is thoroughly cleaned by dipping it into chemically pure ethyl ether, dried in air and immediately introduced into the test vessel.

d) To avoid contamination, the prepared copper shall be handled only with tweezers. The copper wire shall not be re-used.

C-10 DURATION OF TEST

C-10.1 The test-tube shall be introduced into the apparatus previously brought to operational temperature. The duration of the test shall be 164 hours, this being reckoned from the time when the oil reaches a temperature of 100°C and when the flow of oxygen has been adjusted to the prescribed rate of 1 litre/hour. The time taken to adjust the oxygen flow shall not exceed 10 minutes.

C-10.2 The measurements of sludge and neutralization value, after 48 and 96 hours, are carried out only when it is desired to plot an ageing curve.

C-11 DETERMINATIONS ON OXIDIZED OIL

C-11.1 Sludge Formation

C-11.1.1 The sludge shall be precipitated by adhering strictly to the procedure described below.

C-11.1.2 The sample of 25 g of artificially aged oil shall be cooled in the dark for 1 hour, and then poured into an Erlenmeyer flask of 500 ml capacity, fitted with a ground glass stopper. Use 300 ml of n-heptane to recover, by swrilling, the oil adhering to test-tube, copper spiral and oxygen lead-in tube, n-heptane should then be added to the oil in the flask.

C-11.1.3 The mixture shall be allowed to stand in the dark for 24 hours, at a temperature of 27±2°C, before filtering through a glass filter previously dried to constant mass. The maximum diameter of the pores of the glass filter shall be between 5 and 15 microns, when determined in accordance with the method described in **C-11.1.8**.

C-11.1.4 At the start of filtering only a small pressure drop should be used to prevent the sludge passing through the filter. Cloudy filtrates should be passed through a second time.

C-11.1.5 All traces of oil shall be carefully removed by repeated washing of the sludge with n-heptane. The total volume of the n-heptane used for the washing of the sludge shall be 150 ml. The filter containing the sludge is dried at 110°C to constant weight.

C-11.1.6 Sludge adhering to the catalyst, to the test-tube, and to the oxygen lead-in tube is transferred, by dissolving it in small quantities of chloroform (a total of 30 ml), to a tared porcelain vessel. It is then dried at 110°C after evaporation of the chloroform, to constant mass. The weight of the residue is added to that of the sludge obtained by precipitation with normal heptane.

C-11.1.7 The total sludge is expressed as a percentage of the initial mass of the oil.

NOTE — If n-heptane recovered from previous tests is used, it is important that its acidity be checked before reuse. A product which is not neutral shall be purified before use.

C-11.1.8 *Determination of Maximum Diameter of Pores of a Glass Filter*

It is not easy to determine the mean diameter of the pores of a glass filter. The determination of the maximum diameter of the pores provides a valuable indication of the capacity of the filter, that is the maximum diameter of spherical particle that may pass through it.

The maximum diameter of the pores is determined by measuring the air pressure necessary to cause the first air bubble to pass through the filter moistened by the liquid to be tested.

The following relation is used:

$$D = \frac{30 \times 10^3 \times r}{P}$$

Where

D = diameter in microns of the pores,

r = surface tension of the test liquid in N/m, and

P = observed pressure in mm Hg.

In order to check a filter having a maximum pore diameter of 5 to 15 microns, either water or carbon tetrachloride is used as a test liquid.

The glass filter is completely immersed in the test liquid of which a film of several millimetres thickness will remain on the upper surface of the filter attached to the apparatus (*see* Fig. 2).

A uniformly increasing pressure is applied to the filter until one or two bubbles of air appear in the liquid above the filter. The manometer reading enables the maximum diameter of the pores to be calculated. This manometer may be directly calibrated in pore diameters.

For a filter whose maximum pore diameter is between 5 and 15 microns, the manometer reading in mm Hg will be 435 to 145 for water and 160 to 55 for carbon tetrachloride.

C-11.2 Neutralization Value

C-11.2.1 The heptane solution obtained after filtering off the sludge is collected in a 500 ml measuring flask and made up to the mark with normal heptane. Three determinations of the neutralization value are made on 100 ml samples of the heptane-oil solution.

C-11.2.2 Immediately before use, the titrating solution is prepared as follows:

a) Add 1 to 3 ml of the alkali solution and a drop of 0·1 N HCL to sensitize the indicator to 100 ml of a mixture of 3 parts of benzene to 2 parts of ethyl alcohol by volume.

b) Neutralize the mixture by 0·02 N potassium hydroxide to give a red colour comparable to that of a 10 percent solution of cobalt nitrate [$CO(NO_3)_2\,6H_2O$]. This colour shall persist for at least 15 seconds.

c) Add the same volume of neutralized solvent to 100 ml of the heptane solution while stirring.

This solution is then titrated with 0·02 N alcoholic potassium hydroxide at a temperature not exceeding 25°C.

C-11.2.3 The neutralization value shall be calculated according to the following formula:

$$\text{Neutralization value} = \frac{(n_2 - n_1)\ 56{\cdot}1N}{5}$$

where

n_2 = volume in ml of 0·02 N alcoholic potassium hodroxide solution necessary to neutralize the normal heptane oil solution,

n_1 = volume in ml of 0·02 N alcoholic potassium hydroxide solution necessary to neutralize 100 ml of normal heptane to which have been added 100 ml of the titrating solution,

N = normality of alcoholic potassium hydroxide solution, and

5 = the ratio of total volume of heptane solution of oil to its volume used in the titration.

C-12 REFEREE TESTS

C-12.1 Taking into account the accuracy that may normally be expected from the basic measurements employed during the oxidation stability test, the following requirements apply to referee tests:

a) *Number of Samples* — Referee tests shall be carried out simultaneously in triplicate.

b) *Weight of Sludge* — If the spread between the three masses in relation to the arithmetical mean does not exceed the maximum value shown in the table below, then the test is valid.

Mass of Sludge (Mean)	*Maximum Permissible Spread of Results*
mg	mg
Up to 10	2
Up to 20	4
Up to 40	6
Up to 50	8
Up to 100	10
Above 100	20

The sludge content is the arithmetical mean of the three tests expressed as a percentage.

c) *Neutralization Value Total Acidity* — If the maximum spread between the three results does not exceed 40 percent of their arithmetic 'mean', then the test is valid and this 'mean' is the result.

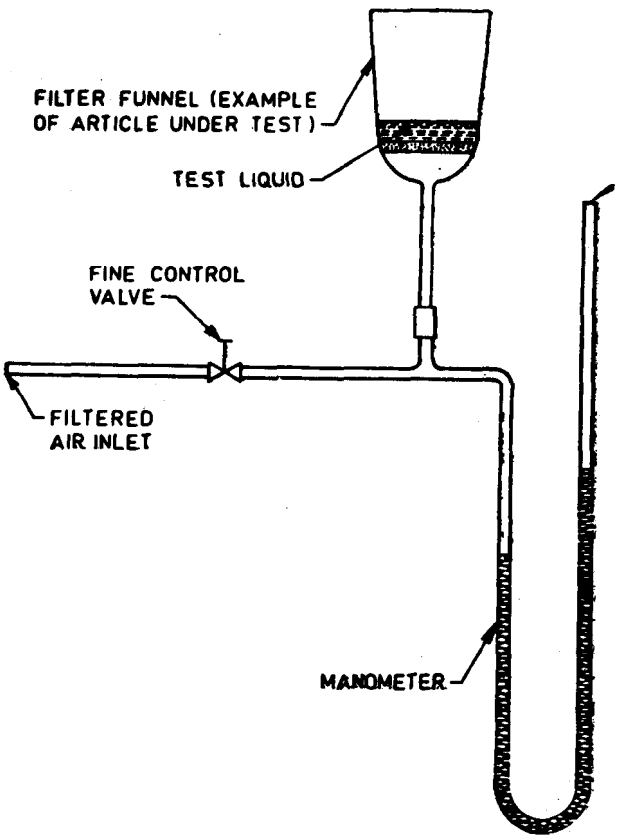


FIG. 2 APPARATUS FOR DETERMINATION OF PORE DIAMETER OF A GLASS FILTER

ANNEX D

[*Table* 1, *Item* (xvi)]

METHOD OF DETERMINATION OF SK VALUE

D-1 GENERAL

D-1.1 The behaviour of insulating oils in the presence of concentrated sulphuric acid furnishes information on the refining degree of mineral oil.

D-2 APPARATUS

D-2.1 Settling Tube

Graduated cylindrical glass tube of 20 ml capacity divided in 1/10 ml (*see* Fig. 3), round-bottomed topped by octagonally capped glass stopper with taper ground joint. The interval between the 20·0 ml graduation and the top rim of the tube shall be 7 cm.

D-2.2 Graduated Pipettes

10 ml delivery pipettes, suitably marked to indicate use (for example, coloured rings). The pipette for sulphuric acid should be fitted with a safety device. When it is suspended in the settling tube its tip should be between the 10·0 and 11·0 ml graduation lines. The volumetric lines on both pipettes shall be at least 100 mm below the top rim of the pipette.

D-3 REAGENTS

D-3.1 Concentrated sulphuric acid with H_2SO_4 content 96 ± 0·1 percent, density 1·828 g/ml. The acid shall be stored in bottles of not more than 300 ml capacity with ground-in stopper covered with ground-on cap.

D-4 PROCEDURE

D-4.1 The oil under test, the sulphuric acid and the test apparatus shall be kept at a temperature of 27 ± 2°C.

D-4.2 Transfer sulphuric acid by means of the pipette into the settling tube without touching its innerwall until lower maniscus edge is in level with the 10·0 ml line of the tube. Then carefully top the acid with 10·0 ml line of dry test oil in the same manner by means of the oil pipette. Oil drying is done suitably by filtering it through Whatman No. 1 or equivalent filter paper at 100°C.

D-4.3 Close the settling tube with the glass stopper. Tilt the tube gently but quickly by 180° fifty times in both directions alternately, allowing it to stay in each vertical position until the insulating oil has entirely run off. Then rapidly invert it. Place the tube vertically in a tall 5-litre glass beaker (330 mm high) filled with water to the 250 ml level. The temperature of water shall be maintained at 27 ± 2°C. Allow the mixture to separate for not more than 3 hours. Note the reading of levels at 15 minute intervals. The separation shall be considered complete when no further change in the level of the boundary layer is found to accrue in two observations made at 15 minute intervals.

D-4.4 The increase in volume of sulphuric acid layer is represented by the difference between the level of boundary layer and the 10·0 ml graduation line.

D-4.5 In the case of oil samples not giving clear separation within 3 hours, dilute the oil sample with an equal volume of standard benzene. Repeat the test with 10·0 ml sample and double the result obtained.

D-5 REPORT OF RESULT

D-5.1 Report the increase in volume of sulphuric acid in percent.

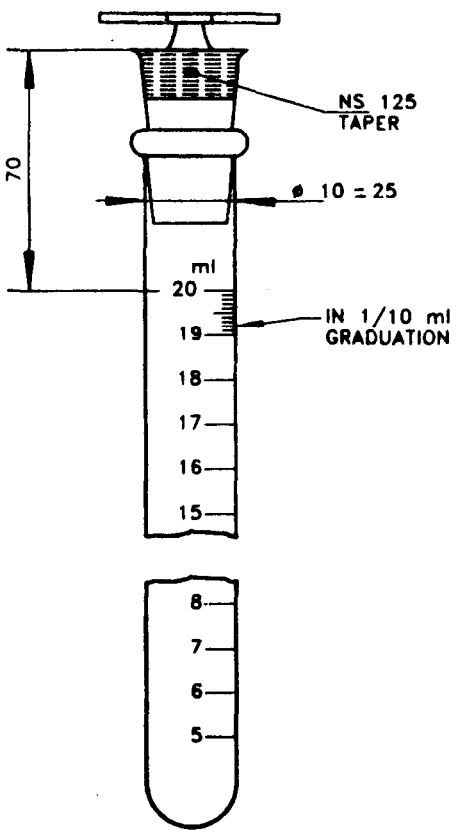


FIG. 3 SETTING TUBE

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TO
IS 335 : 1993 NEW INSULATING OILS — SPECIFICATION

(Fourth Revision)

(*Page* 1, *clause* **1.1**, *line* 4) — Substitute 'arc' *for* 'are'.

(*Page* 3, *Table* 1):

a) *Sl No.* (ii), *col* 4 — Substitute 'IS 1448 [P : 16] : 1990' *for* 'IS 1448 [P :16] : 1977'.

b) *Sl No.* (v), *col* 4 — Substitute 'IS 1448 [P : 21] : 1992' *for* 'IS 1448 [P : 21] : 1970'.

c) *Sl No.* (x), *col* 2, *line* 3 — Substitute '90°C, *Max*' *for* '90°C, *Ma*'.

(*Page* 4, *Table* 1):

a) *Sl No.* (xiv), *col* 2 — Insert 'percent by weight' after 'Presence of oxidation inhibitor'.

b) *Sl No.* (xiv), *col* 3 — Substitute 'The oil shall not contain antioxidant additives' *for* 'The oil shall contain antioxidant additives'.

c) *Note* 3, *line* 3 — Substitute '0.05 (*Max*)' *for* ' 0.5 (*Max*)'.

(*Page* 5, *Annex* A) — Insert the following after '1448 [P :16] : 1990':

'1448 [P : 21] : 1992 Methods of test for petroleum and its products : Part 21 Flash point (closed) by Pensky Martens apparatus (*third revision*).'

(*Page* 5, *Annex* A) — Delete '2832 : 1964 Waterproof silicon carbide paper (withdrawn)'.

(*Page* 5, *Annex* A) — Substitute the following title for IS 6262 : 1971:

'Method of test for power factor and dielectric constant of electrical insulating liquids.'

(*Page* 6, *Annex* B, *clause* **B-3.4**, *line* 2) — Delete '(IS 2832 : 1964)'.

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